Wear behavior and electrical conductivity study of Cu-graphite MMC prepared by powder metallurgy route

A thesis submitted in partial fulfillment of the
requirements for the degree of

Master of Technology
in

Metallurgical and Materials Engineering

by

Jyoti Singh Parihar
Roll No. 211MM1365

Department of Metallurgical and Materials Engineering
National Institute of Technology Rourkela
Rourkela, Orissa-769008

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Under the guidance
of
Prof. D. Chaira

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CERTIFICATE

This is to certify that the project entitled, "**Wear behavior and electrical conductivity study of Cu-graphite MMC prepared by powder metallurgy route",** submitted by **Jyoti Singh Parihar** (211MM1365) in Department of Metallurgical and Materials Engineering at National Institute of Technology, Rourkela is an authentic work carried out by her under my supervision and guidance.

To the best of my knowledge, the matter embodied in the report has not been submitted to any other University/ Institute for the award of any degree or diploma.

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Rourkela

Declaration

I certify that

a) The work contained in the thesis is original and has been done by myself under the general supervision of my supervisor.

b) The work has not been submitted to any other Institute for any degree or diploma.

c) I have followed the guidelines provided by the Institute in writing the thesis.

d) Whenever I have used materials (experimental analysis, and text) from other sources, I have given due credit to them by citing them in the text of the thesis and giving their details in the references.

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Date: **Jyoti Singh Parihar**

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Abstract

Copper is most commonly used material in electrical contacts as it possesses high electrical and thermal conductivity and low thermal expansion co-efficient. But the problem is that it has low wear resistance and poor mechanical properties. In the present investigation, our main aim is to improve wear resistance and mechanical properties of copper-graphite metal matrix composite with minor loss of electrical conductivity.

Here, we have studied the effect of pitch coke and milling on wear resistance and electrical conductivity of Cu-graphite composite. Copper-graphite metal matrix composites containing 1, 3, 5 and 10 vol. % of graphite plus pitch coke were prepared by conventional powder metallurgical route where graphite/pitch coke ratio are 50:50(by weight) and 30:70 (by weight). The composite powder mixture were cold compacted and sintered in tubular furnace at 900° C for 1h with argon gas. The composites were then characterized by x-ray diffraction (XRD) and scanning electron microscopy (SEM). It was observed that high hardness values of Cu-graphite metal matrix composite with graphite/pitch coke ratio of 30:70 (by weight) as compared to 50:50. It has also been found that there is not much improvement on hardness due to soft nature of graphite. Wear depth decreases with increasing graphite content as graphite acts as a lubricating film during wear on the contact surfaces. It has also been noticed that MMC with graphite/pitch coke ratio of 30:70 shows higher wear resistance due to higher hardness as compared to 50:50. Wear rate and wear volume was calculated and found that it decreases with increase in vol. % of graphite. It has also been found that higher wear resistance for Cu-graphite MMC of 30:70 graphite/pitch coke ratio as compared to 50:50 because of high hardness. That leads low penetration depth of indenter on sample resulting high wear resistance. Electrical conductivity analysis shows decrease in electrical conductivity with increase in vol. % of graphite plus pitch coke because number of grain boundaries increases as a result of increase in reinforcement content and provide obstacles to electron movement hence increase in electrical conductivity.

To study the effect of milling, Cu with 1 & 5 vol. % of graphite powders were milled for 2, 4, 8, 20 and 40h. Milling was conducted in Pulverisette-5 planetary ball mill under toluene to prevent oxidation. The milled powder mixture were cold compacted and sintered in tabular furnace at 900° C for 1h with argon gas. It can be seen from the micrographs that initially

there is an increase in particle size (flake formation) due to ductile nature of Cu. But in later stage reduction in particle size takes place due to strain hardening during milling. In initial milling period (up to 10 h) hardness trend is decreasing but after that hardness value goes up. The reason is large porosity and less density in the initial milled samples and later porosity decreases and density increases. After 40 hours of milling, the particle becomes fine and more closely bound and particle-particle contact increases with increase in fineness due to increase in surface area of the milled powder particle. Milling of initial Cu and graphite powder mixtures result in higher wear resistance than un-milled powder. Wear rate and wear volume decreases with increase in milling time because of increase in wear resistance and high hardness of Cu-graphite MMC (milled) as compared to (un-milled). Electrical conductivity decreases with increase in milling time as milling introduces number of defects and impurity.

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Chapter 1

Introduction

1 Introduction

1.1 Background

Today our modern technologies require materials with unusual combinations of properties that cannot be met by the conventional ceramics, polymeric materials and metal alloys. This is true, especially for the material needed in transportation, aerospace and under water applications. These combinations of material properties are achieved by the development of composites [1].

Today the numerous features of composite led to the frequently adaptation of it, in the field of engineering. These materials fulfill the demand of almost all engineering application due to their tremendous physical and mechanical properties i.e. light weight, high strength, improved density and hardness, high wear and high corrosion resistance [2].

Copper is used as industrial and functional metal due to its electrical and thermal conductivity. During the operation of large scale electrical machinery the main problem is to transfer the current from one conductor to another which is achieved by sliding one conductor over other. Current transfer takes place through contact surfaces. This type of contact surfaces impose conflict between the requirements because one hand we need lager contact force for effective current transformation and another hand we require low contact of surfaces to reduce wear rate of sliding contact. We cannot satisfy both the requirement of sliding contact individually.

Copper–graphite metal matrix composites are widely used as contact strips for pantographs and collector shoes in electric railways and brushes for motor technology nowadays, which combine the properties of copper, i.e. thermal conductivities and super excellent electrical, properties of graphite, i.e. solid lubricating, a low friction coefficient and small thermal expansion coefficient. In recent years, it is strongly required to quicken up trains and reduce costs to maintain the facilities for the fast development of electric railway technology. However, high running speed causes an increase of contact break and wear rate between strips of current collector and trolley wires easily, increasing cost to maintain the facility and reducing the life of contact strip and wires. Therefore, in order to meet these requirements, it is necessary to improve the mechanical and tribological properties of contact strips. As well known, graphite is excellent solid lubricant. Consequently, the addition of graphite is effective to increase the wear resistance and decrease in the friction coefficient with minor

loss in electrical conductivity [3]. So our present work is concentrated on the effect of pitch coke addition and milling on wear and electric conductivity of Cu-graphite MMC's prepared by powder metallurgy route.

1.2 Objective of the project

1. Fabrication of Cu-graphite metal matrix composite with 1, 3, 5 and 10 vol. % of graphite.
2. Effect of pitch coke on Cu-graphite MMC, where the graphite/pitch coke is 50:50 and 30:70 (by weight).
3. Study the effect of milling time on the Cu-graphite MMCs.
4. Improvement of wear resistance of Cu-graphite MMCs.
5. Improvement of mechanical and physical properties of Cu-graphite composite.
6. Electrical conductivity study of Cu-graphite MMCs.

1.3 Scope of the project

This thesis includes seven chapters. First chapter explains the background of composite, properties and applications. A brief of metal matrix composite, its processing route and literature review is presented in second chapter. Third chapter elaborates the detailed experimental parameters and procedure adopted. Results and discussions are briefly enumerated in chapter four. Conclusion or overall findings of our experiments is described in chapter five. Chapter six and seven present the future scope of our work and references.

Chapter 2

Literature review

This chapter summarizes introduction and detailed overview about metal matrix composite, its processing routes and applications areas. Also provide background to the present study.

2 Literature review

2.1 Introduction

Composites are combinations of two or more different material, combined in such a way that makes better use of its constituent elements. Composite provides better mechanical, chemical and physical properties in contrast with mechanical alloys in which the constituent element retain their individual properties. Matrix and reinforcement are two constituent elements in composite. Matrix is a continuous phase i.e. polymer, metal and ceramic. Polymer has low stiffness and strength as compared to metal matrix composite and metal matrix composite has high ductility and low strength as compared to ceramic matrix composite. To hold the imbedded phase in place and share the load with the secondary phase is the purpose of matrix material. The selection of matrix is depended upon the type of composite application. In most cases reinforcements or imbedded phase i.e. glass, carbon, organic, boron, ceramic and metallic fibers are stronger, stiffer and harder than matrix. In terms of composites, complete rethinking of an established design is very cheaper and better solution because of ease in the manufacturing of complex shapes [4]. Composites can be classified as:

1. Metal matrix composite (MMC)
2. Polymer matrix composite (PMC)
3. Ceramic matrix composite (CMC)

2.2 Metal matrix composite

Metal matrix composite is in-comparatively a new way of strengthening metals. It is seen along with the other strengthening processes like rapid solidification rate (RSR), mechanical alloying and splat coating. Non-equilibrium microstructures are produced by all of these techniques. Metal matrix composite is different from these techniques in term of strength and stiffness [5]. In metal matrix composite, matrixes are metal and its alloy. The characteristic of these materials depends on the volume fraction of the reinforcement, two constituent elements and manufacturing process in terms of mechanical and physical properties. By this it is possible to get high strength, high wear and corrosion resistance, high fatigue and creep resistance as compared to base material. Metal matrix composites are classified on the basis of reinforcement used and its characteristics:

(1) Continuous reinforcement composite (continuous fibers and filaments)-

(2) Dis-continuous reinforcement composite (whiskers, short fibers and particles)

(a) (b) (c)

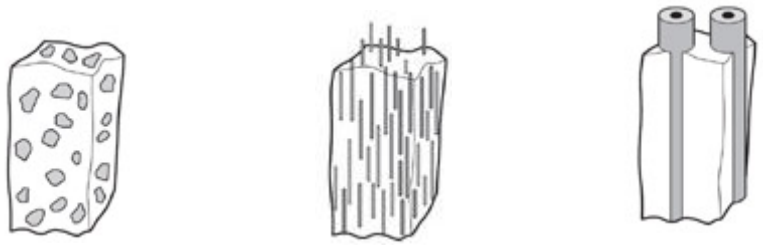


Figure 2.1 Schematic representation of the reinforcement type of composite (a) particle reinforcement (b) Short fiber or whiskers reinforcement (c) long fiber reinforcements [4]

2.3 Processing route of metal matrix composite

Metal matrix composite can be prepared by numbers of processing techniques. The selection of suitable processing technique depends upon application area, quality, and distribution of reinforcement. It is possible to obtain different characteristics profile of composite by altering the manufacturing, processing and finishing method even when same amount and composition of components are concerned. Metal matrix composites can be fabricated by both solid state and liquid state processing. The different solid state processing techniques are described below.

2.3.1 Liquid- state processes

Infiltration of particulate or fiber reinforcement is involved in casting or liquid infiltration preform by liquid metal. Liquid phase infiltration is not straightforward, because of difficulty in wetting of the reinforcement. When infiltration of fiber occurs, reaction takes place in-between the molten metal and reinforcement which degrade the properties of fibers. To control interfacial reaction and to improve wetting fiber coating are applied prior to infiltration. Surface oxidation of the coating is the drawback of fiber coating when exposed to environment. Duralcan process is quite successful in which ingot grade aluminum and ceramic particles are mixed and melted. At slightly above the liquidus temperature (600-700 °C) melt is stirred. Solidified ingot may go through secondary process i.e. extrusion and rolling. Duralcan process of making particulate composite involves use of particles size (8-12μm) [6].

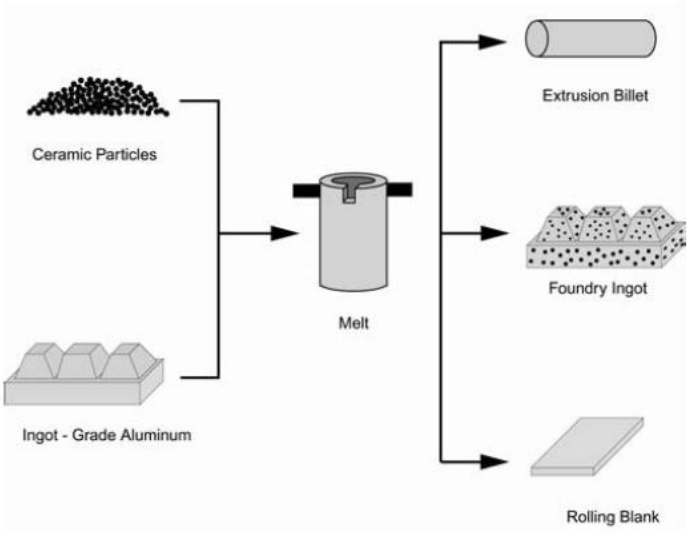


Figure 2.2 Casting process [6].

2.3.2 In-situ processes

The reinforcement phase is formed in-situ in this process. The composite material is prepared is one step starting from appropriate alloy therefore this technique avoids the difficulty i.e. combination of two different phases which is present in all of processing methods. Controlled unidirectional solidification of eutectic steel is a common example of in-situ process.

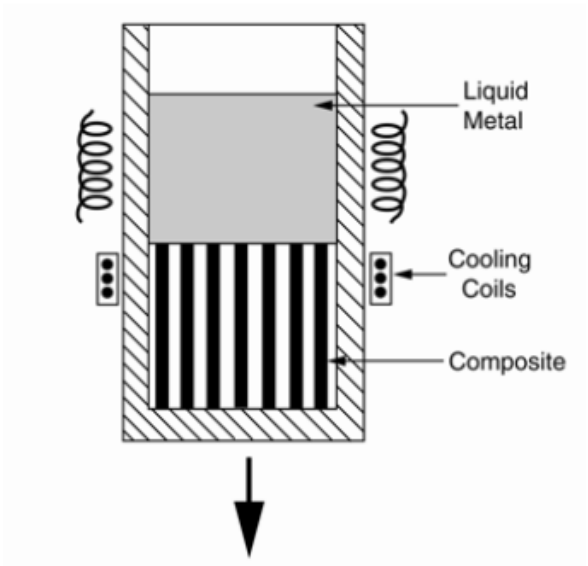


Figure 2.3 In situ process [6].

Controlling the solidification rate leads to control of size and spacing of the reinforcement phase. XD process is one another type of in-situ process which uses the exothermic reaction between two components to prepare third component. Generally used for preparation of composite with large volume fraction [6].

2.3.3 Spray-Forming of Particulate MMCs

Particle-reinforced MMCs is prepared by using modified spray forming techniques also used some times for preparing monolithic alloy. Co-spray process is one particular example of this technique.

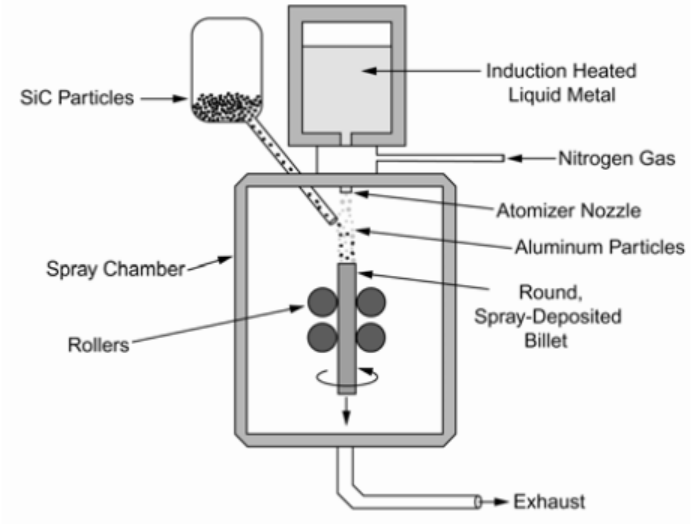


Figure 2.4 The spray-forming process [6].

Spray gun is provided to atomize molten matrix material i.e. aluminum. Heated silicon carbides are injected into atomized aluminum. Quite porous preform is produced. Scalping, consolidation, and secondary finishing processes are applied in co-spray technique to form wrought composite. Aspect ratio of 3-4 and up to 20% volume fraction reinforcement i.e. Silicon carbide particles have been incorporated into aluminum alloy. This process provides flexibility in preparing different type of composites. This process is somewhat expensive, mainly because of the costly capital equipment [6].

2.4 Solid state processing

2.4.1 Diffusion bonding

Diffusion bonding is very common technique for joining of similar and disses similar materials. At higher temperature the interdiffusion of atom between metallic surfaces leads to bonding. Control of fiber orientation, volume fraction and ability to process wide range of metallic material are the principle advantages of this technique. High processing pressure and

temperature, long processing time are disadvantages of diffusion bonding. There are numbers of invariants in solid state processing. Vacuum hot processing is important step in diffusion bonding. In diffusion bonding first step is to apply metal foil than cut into desired shape. Heat is applied to plied followed by lay-up of desired plies. Apply High pressure and hold for consolidation. Than cool, remove and clean the part [6].

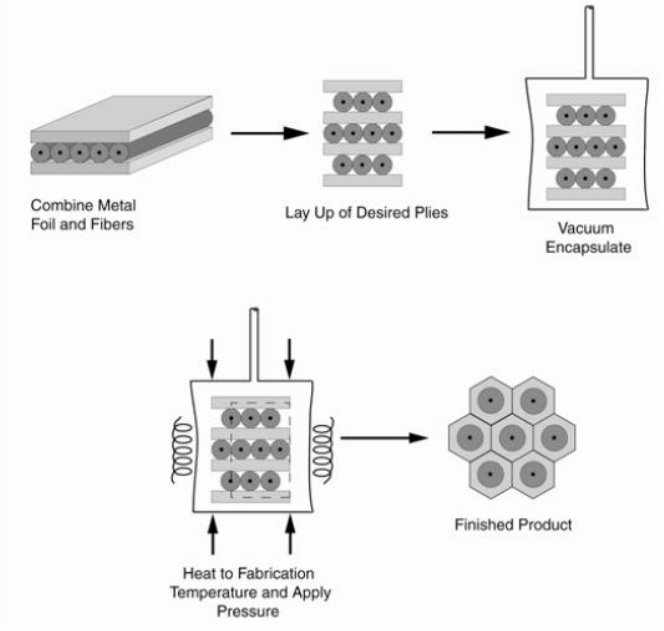


Figure 2.5 Diffusion bonding process [6].

2.4.2 Powder processing

Plenty of metal, metal oxides and non-metallic materials are present with desired properties i.e. wear and corrosion resistance but low conductivity. Combination of these materials with copper give required combinations of properties. Powder metallurgy is the only viable method for production of this type of combination at room temperature. Powder processing techniques (i.e. solid state process) is used to fabricate short fiber or particulate reinforced composites. Typically involves blending of reinforcement and matrix powders to get homogenous mixture. Blending stage is followed by cold compaction to produce desired shape which is approximately 80% dense and easy to handle. To produce fully dense body, hot isostaic or uniaxial pressing is applied [6].

2.3.2 Powder metallurgy

Plenty of metal, metal oxides and non-metallic materials are present with desired properties i.e. wear and corrosion resistance but low conductivity. Combination of these materials with copper give required combinations of properties. Powder metallurgy is the only viable method for production of this type of combination at room temperature. Powder processing techniques (i.e. solid state process) is used to fabricate short fiber or particulate reinforced composites. Typically involves blending of reinforcement and matrix powders to get homogenous mixture. Blending stage is followed by cold compaction to produce desired shape which is approximately 80% dense and easy to handle. To produce fully dense body, hot isostaic or uni-axial pressing is applied [6]. Fig. 2.4 shows the flowchart of powder metallurgy technique.

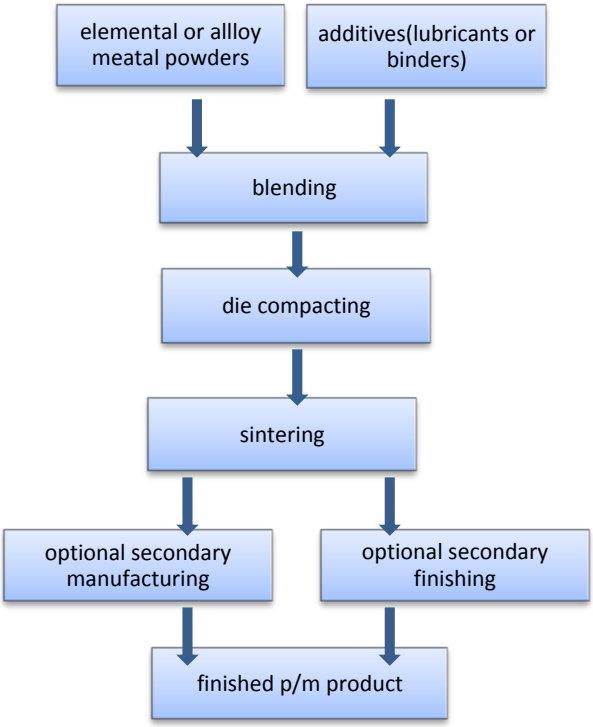


Figure 2.6 Flow diagram of powder metallurgy technique

In powder metallurgy technique, one important step of fabricating composite is consolidation/sintering of green compact. Sintering refers to the process of firing and consolidation of powder at ($T<0.5\ T_m$), where mass transfer by diffusion and leads to the formation of a dense body. The various sintering mechanisms are:

1. Surface diffusion
2. Vapor transport
3. Lattice diffusion from surface
4. Lattice diffusion from grain boundary
5. Grain boundary diffusion
6. Plastic deformation

During sintering, mass transport from one place to another place and two particles are joined together and form a neck. As sintering proceeds neck becomes large by void shrinkage and finally a dense product is formed. Fig. 2.3 shows the formation of neck during sintering.

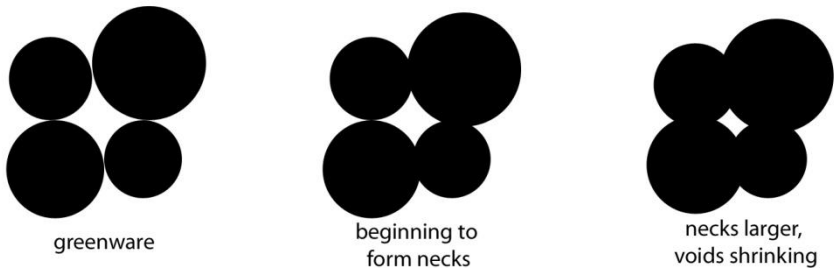


Figure 2.7 Neck formation mechanism during sintering

A brief overview on synthesis of Cu-graphite MMC by powder metallurgy route is presented below. **Yang et al.** explained how the ratio of pitch coke and graphite affects tribological and mechanical properties of copper-carbon composite. They showed that interfacial strength increases with the addition of pitch coke in-between phenolic resin and carbon particle. Mechanical properties increases and attains maximum value with increase in the percentage of pitch coke i.e. micro-hardness, co-efficient of friction and bending strength. Adhesive wear mechanism is present in-between copper-carbon composite, as the percentage of graphite/pitch coke reached to 30:70 wt.%, very slight adhesive wear occur due to the presence of lubricating film [7]. **Queipo et al.** observed that thermal treatment of coal tar pitch improves the composition for processing by removing volatile constituents. The composition and structure of graphite decide the extant of interactions between graphite and pitch coke. Incorporation of pitch coke favors in improvement of compressibility [8]. According to **Moustafa et al.,** three wear regions are present in Cu-graphite composites i.e. low, medium

and severe. Copper–coated graphite exhibits high friction coefficient as compared to uncoated and pure copper composite. Same wear mechanism occurs in both copper-coated and uncoated graphite composites [9]. **Kovacik et al.** observed that wear rate and coefficient of friction of copper coated and uncoated composite increases with graphite volume percentage up to certain critical value and after that the coefficient of friction become independent while further decrease in wear rate [10]. **Akhlaghi et al.** pointed out that aluminum and graphite composite exhibits high wear rate and coefficient of friction for base alloy as compare to both dry and oil impregnated sliding. Wear resistance increases with the increase in graphite percentage. In contrast with base alloy, wear rate and coefficient of friction is always higher in oil impregnated sliding than dry sliding because in dry sliding with the increase in graphite percentage leads to formation of solid thick [11]. **Ma et al.** proposed that during sliding process abrasive wear, adhesive wear are dominant wear mechanism [12]. **Chen et al.** suggested that addition of graphite with low content of h-BN stabilize the properties of Cu-graphite composite and h-BN is helpful in formation of tribo film that increase wear resistance [13]. **Gautam et al.** reported that Cu–4Cr–4G has higher wear resistance as compared to Cu–4Cr–3G, Cu–4Cr–2G, Cu–4Cr and Cast copper. Average coefficient of friction decreases with the increase in normal load. However the graphite containing composite show low average coefficient of friction than other material and this may be attributed to the fact that during dry sliding graphite act as lubricating film and provide low wear rate [14]. **Arami H. et. al.** found that during milling aluminum particles undergo severe plastic deformation. Agglomerates CuO were broken down and the brittle particles were disintegrated into smaller pieces. It was observed that crystalline size decreases with increase in milling time. Crystalline size and lattice strain was estimated to be 50nm and 0.35%, respectively [15]. **Jain et al.** found lowest wear rate as 2.9×10^{-4} mm^3 N^{-1} M^{-1} in highest sliding speed 4.19 m s^{-1} fine-grained Cu-graphite composite [16]. **He et al.** observed the microstructure of carbon-graphite metal matrix composite, which is composed of a graphite islands in a copper matrix, provide solid lubrication with minor loss in electrical conductivity. The layer transfer function of carbon graphite metal matrix composite reduces wear. Special electrical conductive mechanism namely network conduction plays a major role in maintaining the low resistivity of CGCMs [17]. **Guler et al.** concluded that pure copper powder metallurgy sample provides higher electrical conductivity. However when processing has been done on it and 4 wt. % oxide reinforced samples exhibited good electrical conductivity among the oxide reinforced Cu composites prepared via mechanical alloying

[18].**Yusoff et al.** studied Mechanical alloying and sintering of nanostructured tungsten carbide-reinforced copper composite and its characterization and they found that increase in particle size with increase in milling time [19]. **Fathya et al.** noticed that formation of third phase at the interphase of copper alumina composite. Increase in percentage of alumina about 12.5 % and strain rate in copper leads to higher compressive strength. Also concluded that wear rate increases with increase in sliding speed and applied load. The wear of monolithic copper is found more as compare to Nano composites [20].

Chapter 3

Experimental details

This chapter summarizes all the experimental procedures starting from preparation of MMCs to characterization by XRD and SEM, particle size analysis and mechanical and physical properties study i.e. theoretical density measurements, micro-hardness test, wear study, wear rate and wear volume calculation and electrical conductivity measurements.

3 Experimental details

3.1 Synthesis of Cu-graphite composite with pitch coke

Synthesis of Cu-graphite MMCs were performed by powder metallurgy route to study the effect of pitch coke on wear behavior and electrical conductivity study of Cu-graphite MMC's.

3.1.1 Conventional powder metallurgy method

Copper-graphite metal matrix composite with 1, 3, 5 and 10 vol. % of graphite plus pitch coke were prepared by conventional powder metallurgical route where graphite/pitch coke ratio is 50:50 and 30:70 (by weight). Blending was done in a mortar and pestle to ensure the uniform distribution and mixing of Cu, graphite and pitch coke. The composite powder mixture were cold compacted and sintered in tabular furnace with argon gas. The various parameters of compaction and sintering are mentioned in Table 1.

Table 1 Sintering parameters used

Compaction pressure	700MPa
Relaxation time in compaction	2 minutes
Sintering Temperature	900ºC
Holding time	1h
Atmosphere	Argon
Heating rate	5°C/minute

3.1.2 Milling of Cu-graphite powder mixture

Copper with 1 & 5 vol. % of graphite powder were milled separately for 2, 4, 8 20 and 40h. Milling was conducted in Fritsch Pulverisette-5 planetary ball mill. The milled powder mixture were cold compacted and sintered in tabular furnace at 900° C for 1h with argon gas to study the effect of milling. The different milling parameters are represented in Table 2.

Table 2 Milling parameters used

Mill type	**Pulverisette-5 planetary ball mill**
Milling time	2, 4, 8, 20 and 40h
Wet milling	Toluene
Milling speed	300 rpm
Grinding media	
Type	Stainless steel
Ball size (diameter)	15 mm
Ball to powder ratio by weight	10:1
Jar volume	250 ml

3.2 Sample characterization

3.2.1 X-Ray Diffraction (XRD) study

The polished samples were mounted on sample holder of (PAN analytical model: DY-1656) for determination of phases present in Cu-graphite MMC's with variation in volume percentage of graphite plus pitch coke. Diffraction pattern was recorded with scanning range and step size of 20-80° and 2°/min respectively by using CuKα (λ=1.5418°) radiation. The phase identification was done by the 2θ value of the XRD pattern.

3.2.2 Scanning electron microscopy (SEM) study

Scanning electron microscopy (JEOL 6480 LV) was used for microstructural characterization of Cu-graphite sample. Micrographs were taken at 20KV accelerating voltage. The morphology and reinforcement particle-matrix interface was observed from the SEM micrographs. Electron dispersive X-ray (EDX) was used for compositional analysis of the samples.

3.3 Physical properties study

3.3.1 Density measurements

Density of the samples was measured by using Archimedes principle. First, the weight of pellets was measured in air and then the pellets were boiled in water to remove pores. Suspended weight and soaked weight were calculated as the bubble formation stopped. Finally, density was calculated and presented as percentage of theoretical density.

3.4 Mechanical property study

3.4.1 Hardness measurement

Vickers hardness tester (Leco Micro-hardness Tester LM248AT) was used for measurement of hardness of the samples. A load of 0.3kgf was applied for 5 seconds. Minimum 5 measurements were taken at equivalent location for each sample to get consistent results.

3.4.2 Wear study

Ball-on-plate wear tester (Ducom, TR-208 M1) was used to study the wear behavior of Cu-graphite MMC's. Stainless steel ball of 4 mm diameter rotates on Cu-graphite MMC with a speed of 20 rpm for 15 minutes. During the experiment a constant load of 20N was applied and 2 mm track radius was selected. From the wear track on the sample, wear rate and wear volume were calculated using the following equations [16]. All the experiments were carried out at least three times to check the reproducibility of the friction and wear data.

Wear volume (mm^3) = $2\pi \times$ track radius $\times$ track width $\times$ wear depth …… (1)

Wear rate ($mm^3\ N^{-1}\ m^{-1}$) = wear volume / (normal load $\times$ sliding distance)…….(2)

3.4.3 Surface profile study

Veeco DEKTAK 150 surface profilometer was used to measure 2-dimentional surface profile of the samples. Constant force of 0.3mg was applied during the experiment to the stylus and 0.2 mm diamond ball was used as indenter. Sample surface profile was taken from surface to wear track than again wear track to surface. Average surface roughness was also measured.

3.4.4 Electrical conductivity measurement

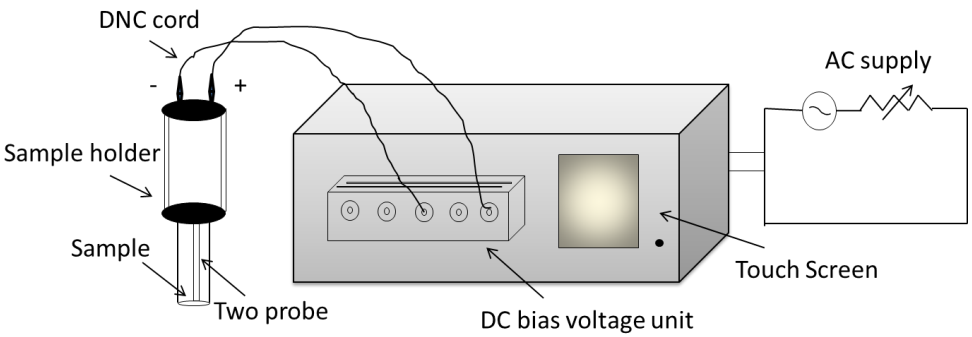


Figure 3.1 Schematic of experimental setup

Electrical conductivity measurement was well conducted on HIOKI 3522-50 LCR HiTESTER at DC voltage with two probe sample holder. The parameters selected for the measurements are impedance (Z), charge electrical density (D), parallel resistance (CP), parallel capacitance (C_{P}). The schematic of the experimental setup used in study was shown in Fig. 3.1. DC bias voltage unit was fixed with the instrument; maximum ± 5 V DC bias was applied during experiment. Sample holder was connected to instrument by DNC cord. Electrical conductivity was measured by formula given below:

$$\boldsymbol{\sigma} = \frac{\mathbf{1}}{\mathbf{R}}\frac{\mathbf{L}}{\mathbf{A}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (3)$$

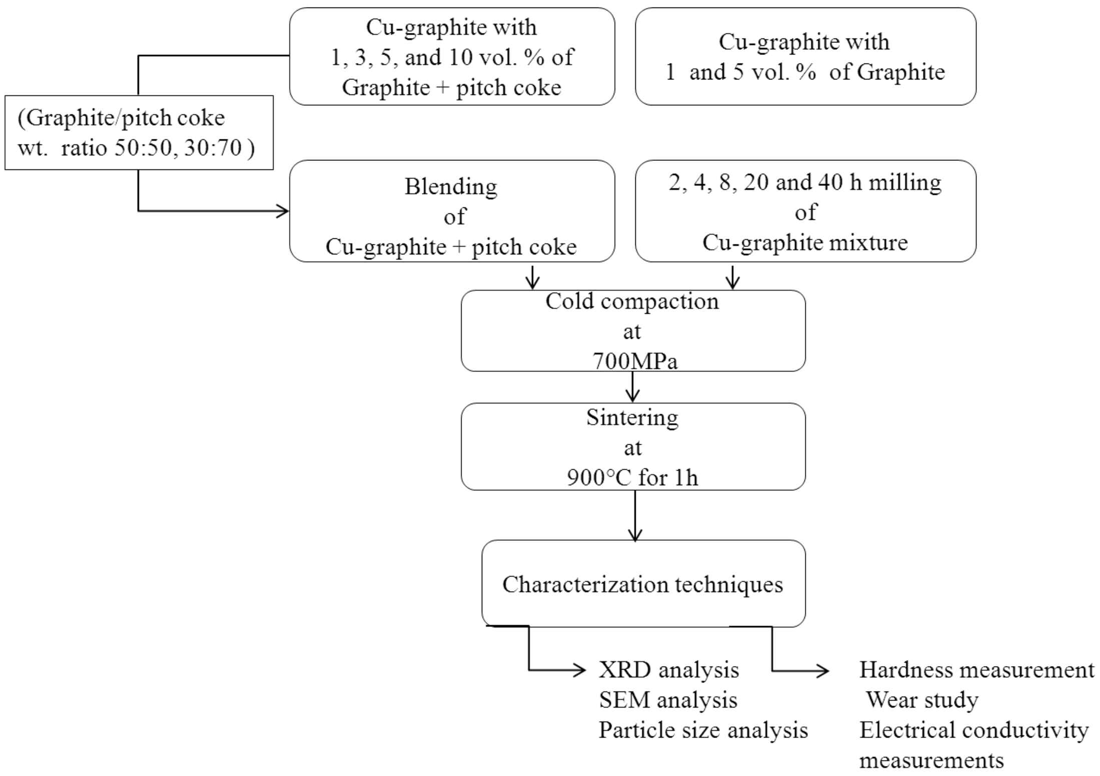
Where, σ = electrical conductivity

R= parallel resistivity

L= sample thickness

A=area of sample

3.4.5 Schematic of over all experimental procedure



Chapter 4

Results and discussions

This chapter includes all the experimental results and analysis of all the data obtained during different experiments i.e. density, hardness, wear study, wear rate and wear volume calculation and electrical conductivity measurements.

4 Results and discussions

4.1 Effect of pitch coke on Cu-graphite MMC's

4.1.1 X-ray diffraction (XRD) study

XRD analysis of copper-graphite MMC with different volume percentage of graphite plus pitch coke was conducted. Fig. 4.1 shows XRD spectrum of Cu-graphite sample sintered at 900°C for 1h with 1, 3, 5, and 10 vol. % of graphite plus pitch coke. XRD spectrum shows presence of strong peak of Cu and very week peak of Cu_2O due to the presence of atmospheric oxygen in the tubular furnace during sintering. It was observed from XRD spectra that during fabrication of composites, no reaction between copper and graphite plus pitch coke takes place. If a new phase is formed during sintering the amount is below the detection level of x-ray diffraction.

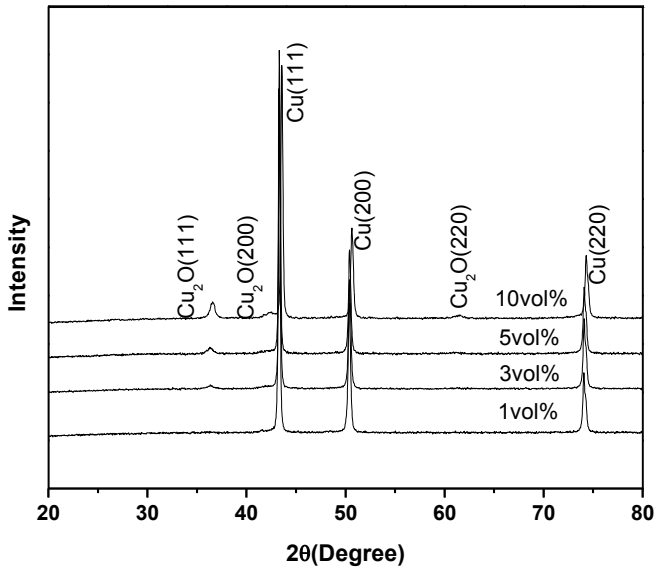


Figure 4.1 XRD spectrum of Cu-graphite MMC's

4.1.2 Scanning electron microscopy (SEM) study

Microstructure of the composites with 1, 3, 5 and 10 vol. % graphite plus pitch coke was studied by the SEM at different magnifications. It was observed from the micrographs shown in Fig. 4.2 that graphite particles (black, reinforcement) are distributed into Cu (matrix). It was also observed from the micrograph that there is more agglomeration of graphite particles in case of Cu-10 vol. % graphite MMC as compared to 5 vol. %. Some of the graphite

particles are found on the surface of composites due to large difference in density between Cu and graphite and low solubility of graphite into Cu matrix. So, proper dispersion of graphite into Cu is a great challenge.

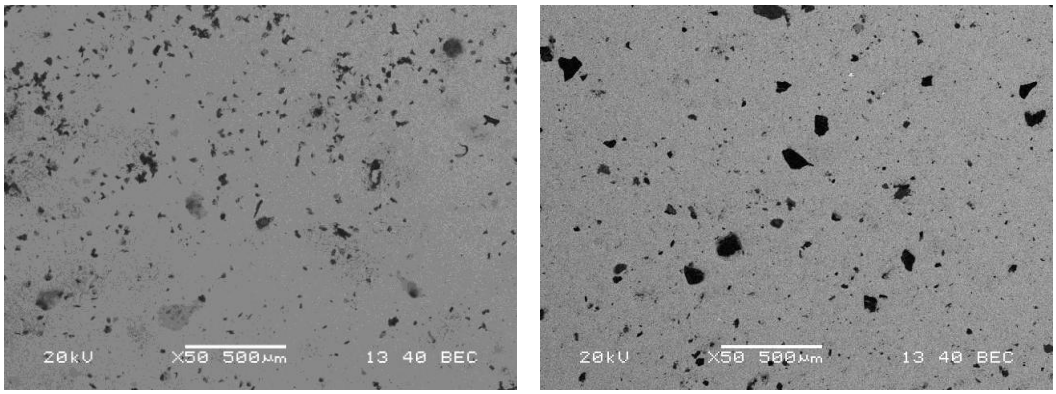


Figure 4.2 Back scattered SEM images of Cu-graphite MMC [5 vol. % (left) and 10 vol. (right)] sintered at 900 °C for 1h

Fig. 4.3 shows EDS spectra taken from whole micrograph of Cu-10 vol. % graphite MMC with graphite/pitch coke ratio (50:50), confirms the presence of copper and small amount of graphite. The quantitative values of copper and graphite were shown in Table 3. Fig.4.4 (a) and (b) shows EDS spectra, captured from light colored (Copper) and dark colored (graphite) respectively.

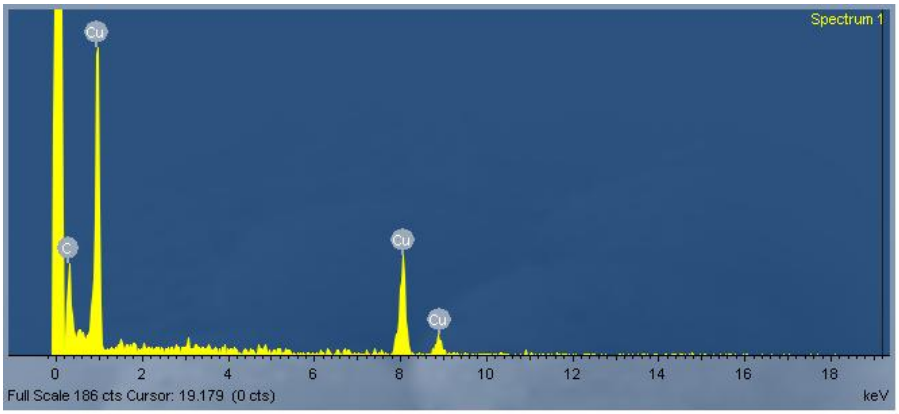


Figure 4.3 EDX spectrum of Cu-graphite plus pitch coke MMC's

Table 3 Quantitative values of EDX spectra captured from whole micrograph

Element	Weight %	Atomic %
Cu	77.97	94.93
C	22.03	5.07
Total	100	

From XRD analysis we concluded that no reaction takes place between Cu-graphite plus pitch coke interface was justified by the EDX analysis. Interface is very clean no interfacial products are present between the interfaces during preparation of composites clearly shown in Fig. 4.4 (b).

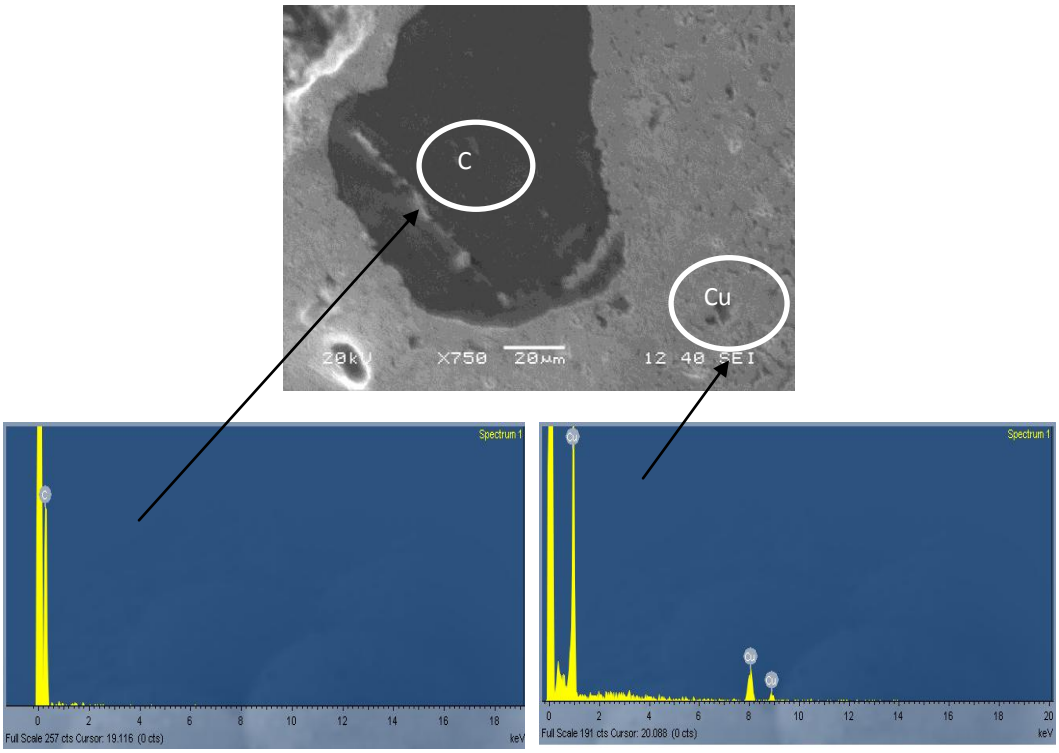


Figure 4.4 (a) Interface between Cu matrix and graphite particles (b) EDX spectra of Cu-graphite MMC's

4.1.3 Densification parameter

Fig. 4.5 shows the variation of densification parameter for 1, 3, 5, and 10 vol. % of graphite plus pitch coke 50:50 and 30:70 graphite/pitch coke ratio and it was observed that densification parameter decreases with the increase in the volume % of graphite plus pitch coke. A reduction in the densification parameter is due to agglomeration and soft nature of graphite. It was also observed that densification parameter for Cu-graphite MMC of 30:70 pitch coke/graphite ratio is less as compared to 50:50. This is due to the presence of pitch coke in higher amount which is porous in nature attributed to low densification parameter. Densification parameter is calculated by using the formula given below:

$$Densification\ parameter\ = \frac{Sintered\ density - Green\ density}{Theoretical\ density - Green\ density} \quad \ldots\ldots\ldots\ldots\ldots (4)$$

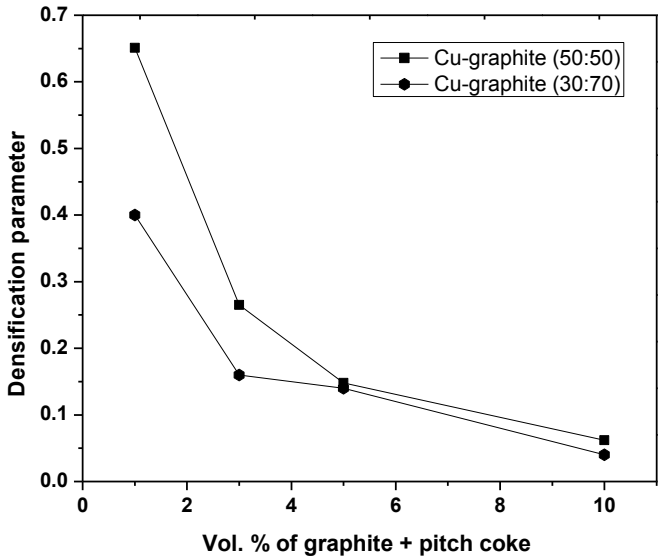


Figure 4.5 Variation of densification parameter with vol. % of graphite +pitch coke

4.1.4 Density measurement

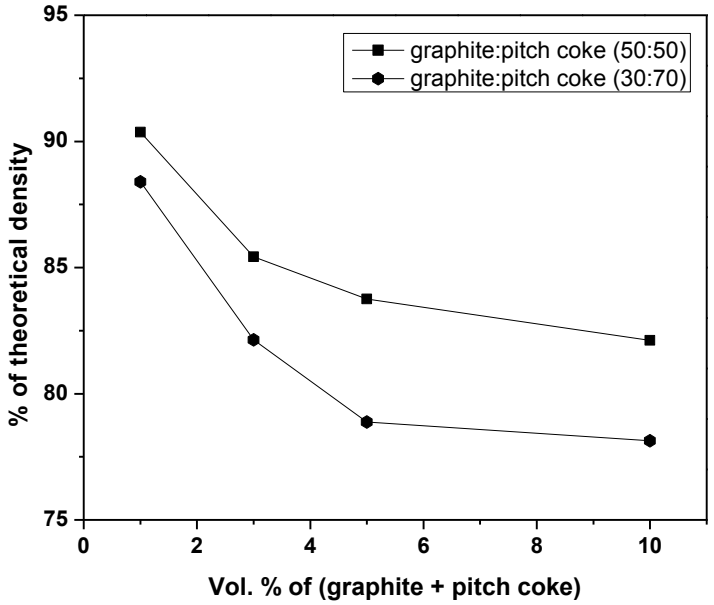


Figure 4.6 Variation of theoretical density with vol. % of graphite plus pitch coke

From Fig. 4.6 we can say that theoretical density decreases with increase in volume percentage of graphite plus pitch coke due to agglomeration and soft nature of graphite. It

also shows density of Cu-graphite MMC with graphite/pitch coke ratio of 30:70 (by weight) is less as compared to 50:50. Lower density of Cu-graphite MMC of graphite/pitch coke ratio 30:70 is due to the presence of higher content of pitch coke. Porosity of pitch coke leads to large number of voids in the sample. Hence porosity of pitch coke attributed to reduction in density of composites.

4.1.5 Hardness study

Fig. 4.7 shows higher hardness values of Cu-graphite MMC's of graphite/pitch coke ratio of 30:70 (by wt. %) as compared to 50:50. It has also been found that there is not much improvement on hardness of composites (with 50:50 wt. %) due to soft nature of graphite. Higher hardness of Cu-graphite MMC of 30:70 graphite/pitch coke ratio is due to the presence of pitch coke. Pitch coke has high porosity, its porosity facilitate the wettability and improves hardness. Yang et al. [3] also studied the effect of the ratio of graphite/pitch coke on the mechanical and tribological properties of copper–carbon composites in which they have also found increase in hardness as content of pitch coke increases and maximum hardness value for a 30:70 wt. % graphite/pitch coke ratio afterward it decreases. They observed higher flexural strength for a sample with 30:70 wt. % graphite/pitch coke ratio which was evident of higher hardness for MMC's with 30:70 wt. %.

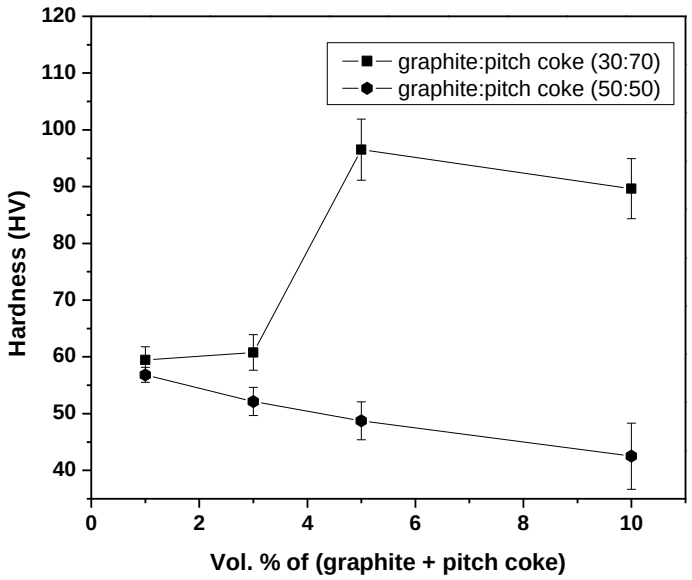


Figure 4.7 Variation of hardness with vol. % of graphite plus pitch coke

4.1.6 Wear study

Fig. 4.8 shows the variation of wear depth with sliding distance for Cu-graphite MMC's and Cu-graphite (pitch coke) 1, 3, 5, and 10 vol. %. From the graph it is observed that wear depth decreases as the vol. % of graphite/pitch coke increases. It was also found that wear resistance of Cu-graphite (with pitch coke) MMC's were higher as compared to Cu-graphite MMC's (without pitch coke) due to the presence of pitch coke. Pitch coke improves the wettability of the composites due to the presence of by porous structure. Pitch coke also provide strong interfacial bonding, act as a lubricating film during wear to prevent direct contact between sample and block. Chen et al. [13] studied tribological properties of solid lubricants (graphite, h-BN) for Cu-based P/M friction composites they observed that wear rate significantly decreases with increase in graphite content. Graphite can form relatively compact tribo-film than H-BN in the wear process; these compact solid lubricant tribo-films are more helpful to improve wear resistance of Cu-graphite composite.

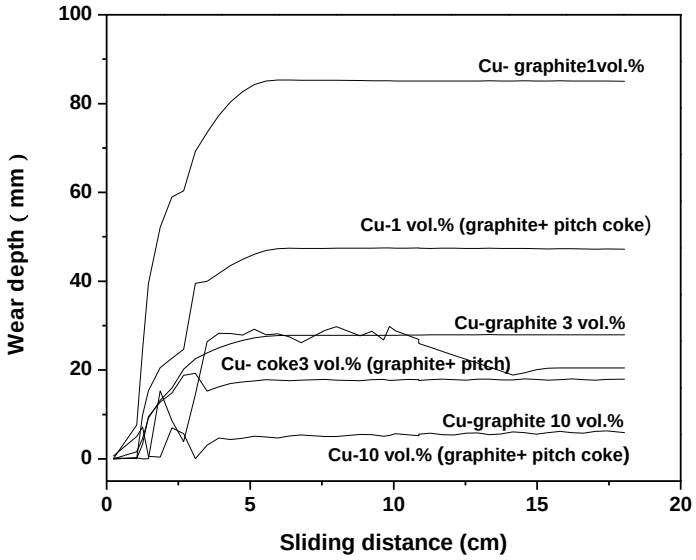


Figure 4.8 Variation of wear depth with sliding distance

Fig. 4.9 shows variation of cumulative wear depth as a function of sliding distance. Maximum wear resistance was found for Cu-10 vol. % graphite where graphite/pitch coke ratio is 30:70 (by wt. %) as compared to 50:50. The wear resistance of the composites increases with increasing the amount of pitch coke. Yang et al. [3] also showed that wear resistance increases with increasing pitch coke and was maximum for 30:70 ratio and afterwards wear resistance decreases. Another reason of higher wear resistance is due to

higher hardness in the composite containing graphite/pitch coke ratio of 30:70. Higher amount of pitch coke leads to the formation of a lubricating film and eventually increases the wettability of the composites and provide strong bonding. Parida et al. [22] explained decrease in wear rate as content of TiO_2 increases in the Ni+TiO2 composite coating surface.

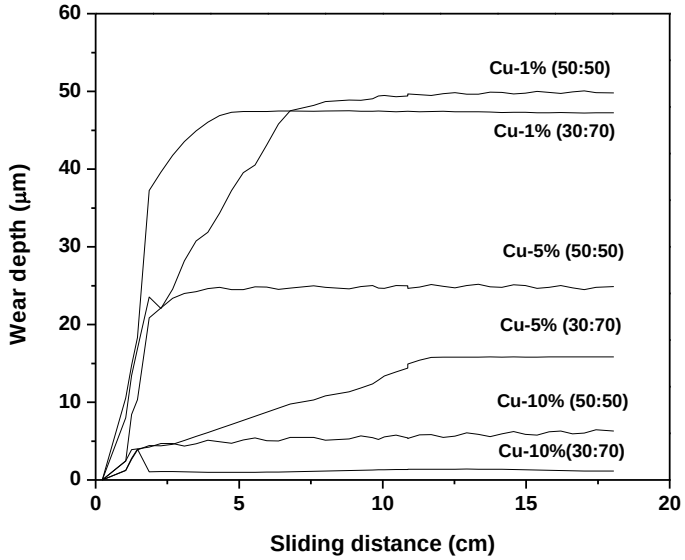


Figure 4.9 Variation of wear depth with sliding distance for Cu-1vol. % and Cu-5 vol. % graphite MMC showing the effect of pitch coke

Fig. 4.10 shows the plot of wear depth vs. sliding distance for Cu-5 vol. % composite (graphite/pitch coke ratio 50:50) at different loads. It is observed from the graph that wear depth increases with increasing load due to soft nature of graphite. Wear of a material is directly proportional to applied load, sliding distance and inversely proportional to hardness. Moustafa et al.[2] studied wear behavior of copper–graphite composites prepared from Cu-coated and uncoated graphite powders and found that three wear regions are present in Cu-graphite composites i.e. low, medium and severe depending on the load applied.

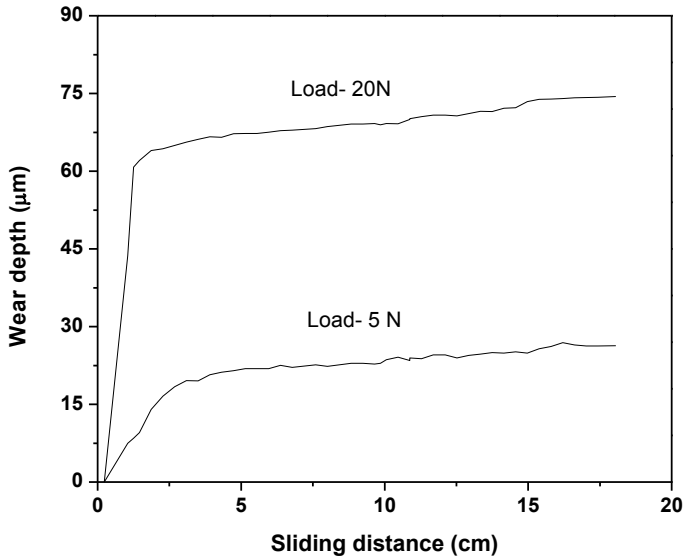


Figure 4.10 Variation of wear depth with sliding distance at different loads for Cu-5 vol. % graphite (graphite/pitch coke ratio 50:50)

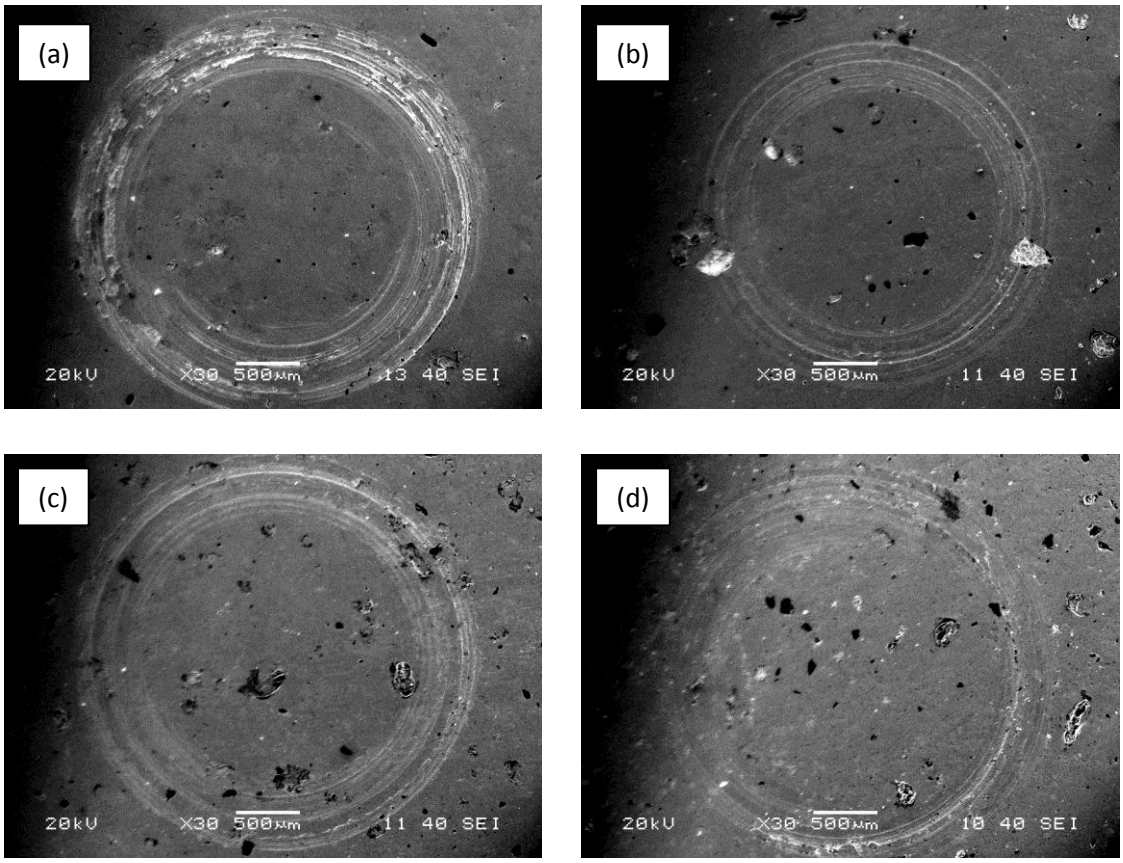


Figure 4.11 SEM images of wear track of (a) Cu-1 vol. % graphite (50:50) (b) Cu-1 vol. % (30:70) (c) Cu-5 vol. graphite (50:50) and (d) Cu-5 vol. graphite (30:70)

Fig. 4.11 shows SEM image of wear track at same magnification for Cu-1 and 5 vol. % graphite composites with different graphite/pitch coke ratios. Fig. 4.11 (a) shows primarily the presence of parallel grooves on worn surfaces represents abrasive wear. Small amount of wear debris are also visible on the wear track. Fig. 4.11 (b) shows plastic deformation of debris (i.e. plough out particles) during wear in the sliding direction leads to adhesive wear. Hence the wear track seems to be smooth for Cu-graphite MMC's with graphite/pitch coke 30:70 as compared to 50:50. Reduction in abrasive wear was attributed to addition of graphite and pitches coke (i.e. reinforcements) as it improves hardness and hence depth of penetration on the surface of sample by the steel ball reduced leading to better wear resistance. These result justifies higher wear resistance of Cu-graphite MMC's with 30:70 wt. % ratio. Jain et al. [16] studied the grain size–wear rate relationship for a-Ti disk in liquid nitrogen environment and they also noticed the presence of grooves and debris initially and in later stage plastic deformation of this debris of wear track leads to increase in wear resistance. Similar observation can also be found for Cu-5 vol. % graphite MMC.

Fig. 4.12 shows the SEM micrographs of Cu-1 vol. % graphite with different pitch coke ratios. The micrographs show that wear mechanism is basically delamination, ploughing of the surface, formation of microcrack and smearing of tribolayer. Another observation can be made from the micrographs that delamination rate is higher in case of 50:50 than 30:70 as the previous is less hard than the later one. Jain el al. [16] also studied grain size–wear rate relationship for a-Ti disk in liquid nitrogen environment they have also found that microcrack, micro-cavity, delamination's during wear on pine on ring ball test.

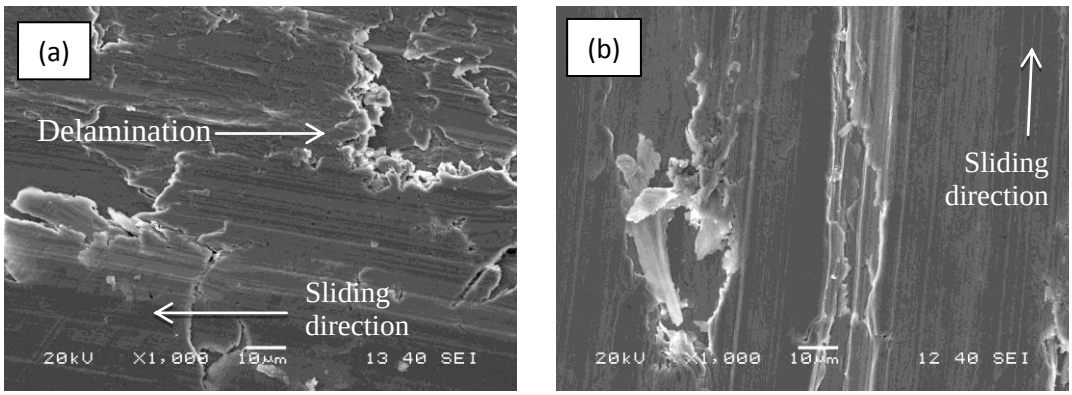


Figure 4.12 SEM image of wear track for (a) Cu-1vol. % graphite (50:50) and (b) Cu-1 vol. graphite (30:70) at higher magnification

4.1.7 Wear volume and wear rate

Wear volume and wear rate were calculated by measuring wear track width, wear depth and sliding distance. Fig. 4.13 and Fig. 4.14 show the wear volume and wear rate plot for Cu-graphite composites (with pitch coke) respectively. It was observed that wear volume and wear rate decreases with increase in volume percentage of graphite/pitch coke. Also it was found that wear volume and wear rate was lesser for MMC's with graphite/pitch coke ratio 30:70 as it provides higher wear resistance as mentioned in Fig. 4.11 and Fig. 4.12. Jain et al. [16] found wear rate for Ti-Al composite of 2.9×10^{-4} $mm^3\ N^{-1}\ M^{-1}$ at highest sliding speed of 4.19 m s^{-1}.

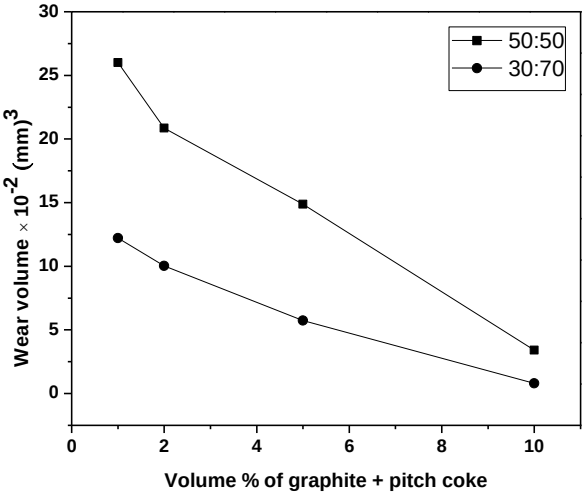


Figure 4.13 Variation of wear volume with vol. % of graphite +pitch coke

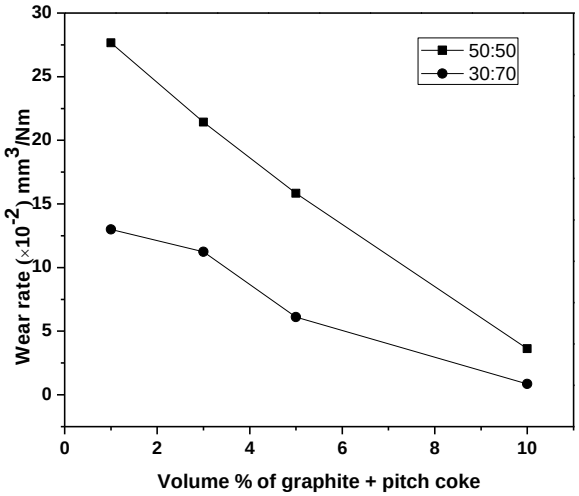


Figure 4.14 Variation of wear rate with vol. % of graphite +pitch coke

4.1.8 Surface profile study

Fig. 4.15 shows the surface profile of Cu-graphite metal matrix composite prepared by powder metallurgy route. The surface profile is taken from surface-wear track-surface of the Cu-graphite MMC. The profile shows the highest variation in wear depth on wear track as compared to surface of the specimen.

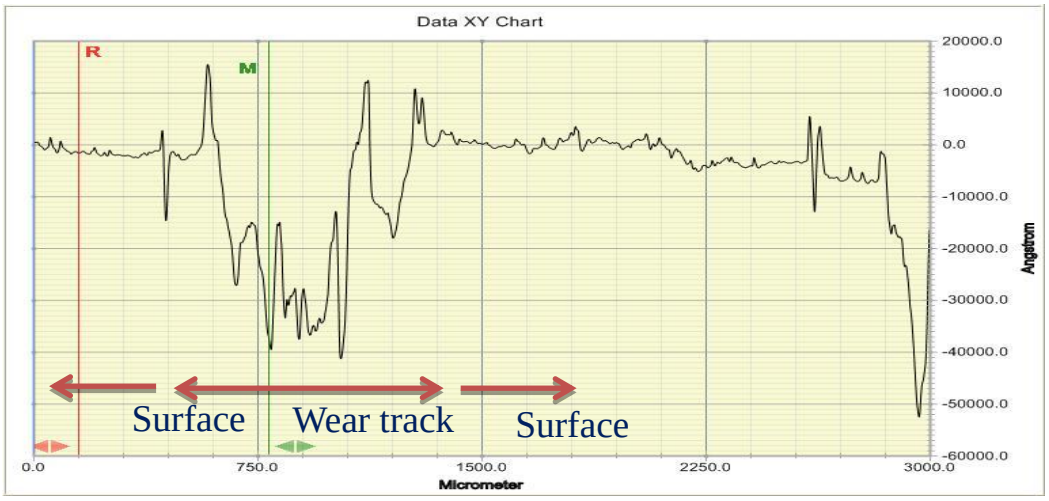


Figure 4.15 Surface profile of Cu-5 vol. % graphite MMC's

Fig.4.16 shows surface roughness increases with increase in volume percentage of graphite this is because of higher wt. % of pitch coke and graphite and there low solubility into Cu matrix which is also seen from fig. 4.15.

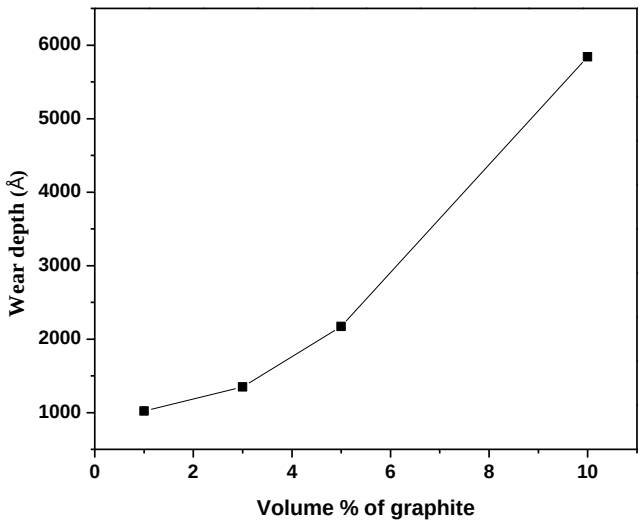


Figure 4.16 Variation of surface roughness with vol. % of graphite/pitch coke

4.1.9 Electrical conductivity measurements

Fig. 4.17 shows the relationship between electrical conductivity of Cu-graphite MMC's with 1, 3, 5 and 10 vol. % of graphite plus pitch coke. It was noticed that electrical conductivity decreases with increase in vol. % of graphite and pitch coke. This is because with increase in graphite and pitch coke content increases the number of grain boundaries and provide obstacles to flow of electrons. This leads to increase in resistivity and consequently decrease in electrical conductivity. Guler et al. [18] investigated contact performance of oxide reinforced copper composite via mechanical alloying; they found that decrease in electrical conductivity as a result of mechanical alloying. Best electrical conductivity was recorded for 4 wt. % ZnO reinforced sample.

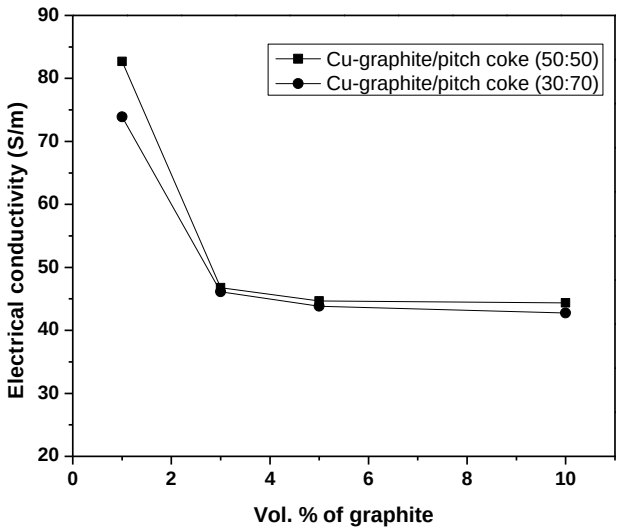


Figure 4.17 Variation of electrical conductivity as function of vol. % of graphite

4.2 Effect of milling

In order to study the effect of milling on initial powder mixture, milling was conducted. Powder mixture of Cu-1 vol. % graphite and Cu-5 vol. % graphite were milled under toluene for 40 hours. Then the powders were cold compacted and sintered in tubular furnace at 900°C for 1 hour under nitrogen atmosphere. Finally the composites were characterized by using different characterization techniques.

4.2.1 X-Ray diffraction (XRD) study

Fig. 4.17 shows the XRD spectra of Cu with 1 & 5 vol. % of graphite powders milled for 2, 4, 8, 20 and 40h. Milling was conducted in planetary ball mill. XRD spectrum shows the presence of strong peak of Cu and very week peak of Cu_2O due to the presence of atmospheric oxygen in the tubular furnace during sintering and with increase in milling time peak intensity of Cu_2O decreases. It can be seen that peak width increases with increasing milling time. During milling powder particles are plastically deformed due to the collision between balls and powders. This indicates that grain refinement takes place and lattice strain increases as milling progresses [21].

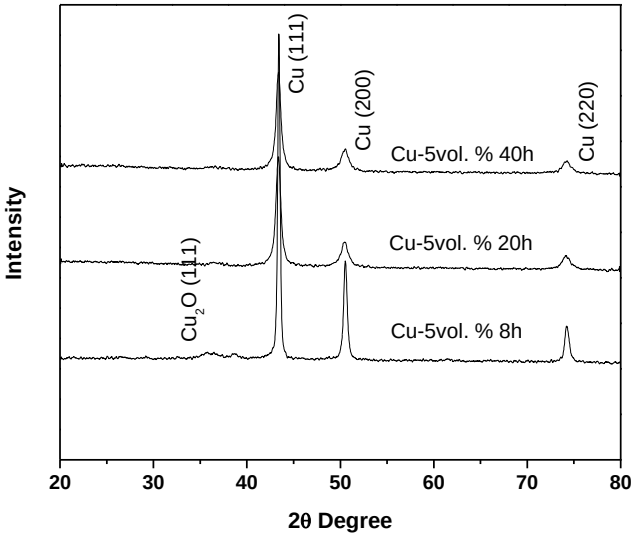


Figure 4.18 XRD spectrum of Cu-graphite MMC's with different milling time

4.2.2 Scanning electron microscopy (SEM) study

Fig. 4.18 shows the SEM micrographs of Cu-1 vol. % graphite composite powder mixture milled for different periods in planetary mill. It is clear from the micrographs that particle size increases up to 20 hours of milling. As copper is ductile, particle size increases in the initial milling period due to flake formation. It is also observed from the micrographs that after 20 hours of milling, size reduction takes place due to strain hardening during milling. It has been noticed that particle shape also changes from flake shape to irregular shape in this stage [21]. Guel et al. [23] also studied the effect of metallic addition on mechanical properties in an aluminum–graphite composite synthesized by means of mechanical milling.

They observed that after 8 h of milling, particles are smaller and rounded and homogeneously distributed into the layers of aluminum matrix.

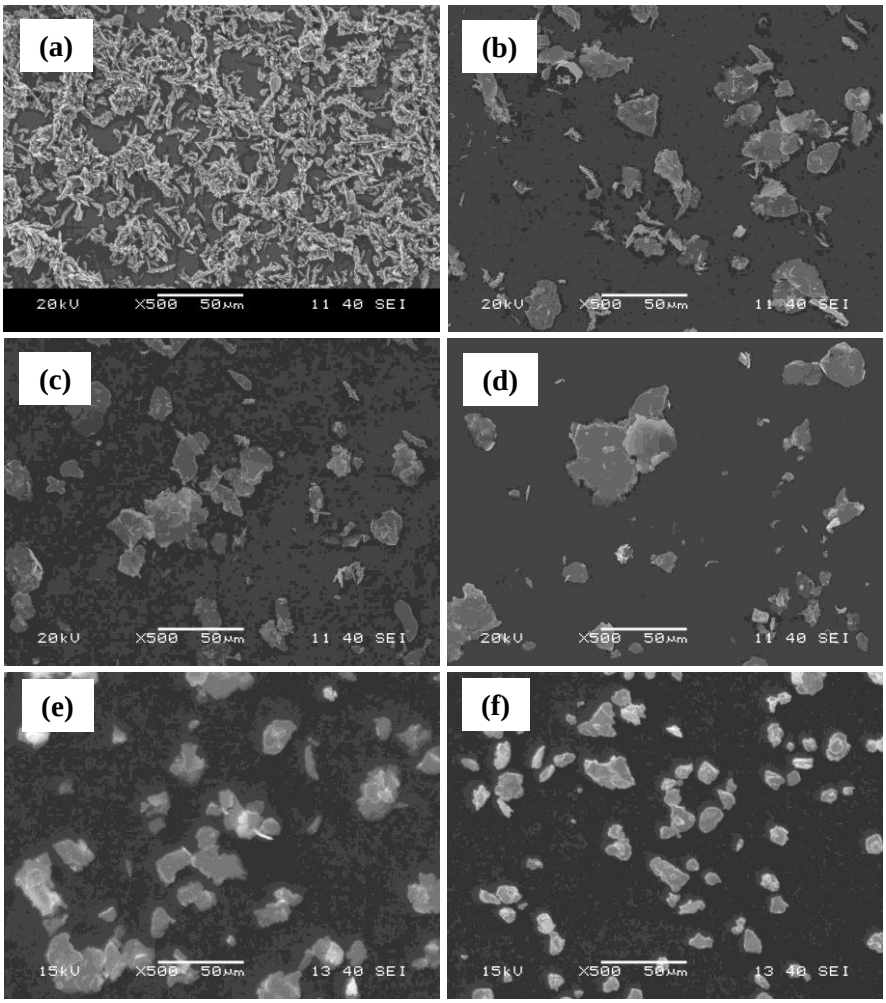


Figure 4.19SEM micro-graphs of Cu-1 vol. % MMC's of powder particles with variation in milling time (a) 0h (b) 2h (c) 4h (d) 8h (e) 20h (f) 40h

Fig. 4.19 shows the SEM micrographs of Cu- 1 vol. % graphite reinforced composite powder mixture milled for different time periods and then sintered at 900°C for 1 hour. The micrographs show the presence of large amount of pores in initial milling period. As the particles being flaky, compaction and sintering of such powder leads to generation of large amount of porosity. But after 20 hours of milling, porosity decreases drastically and it is clearly visible from the micrographs. After 20 hours of milling, particle size changes from flaky to irregular shape and eventually leads to decrease in porosity [21].

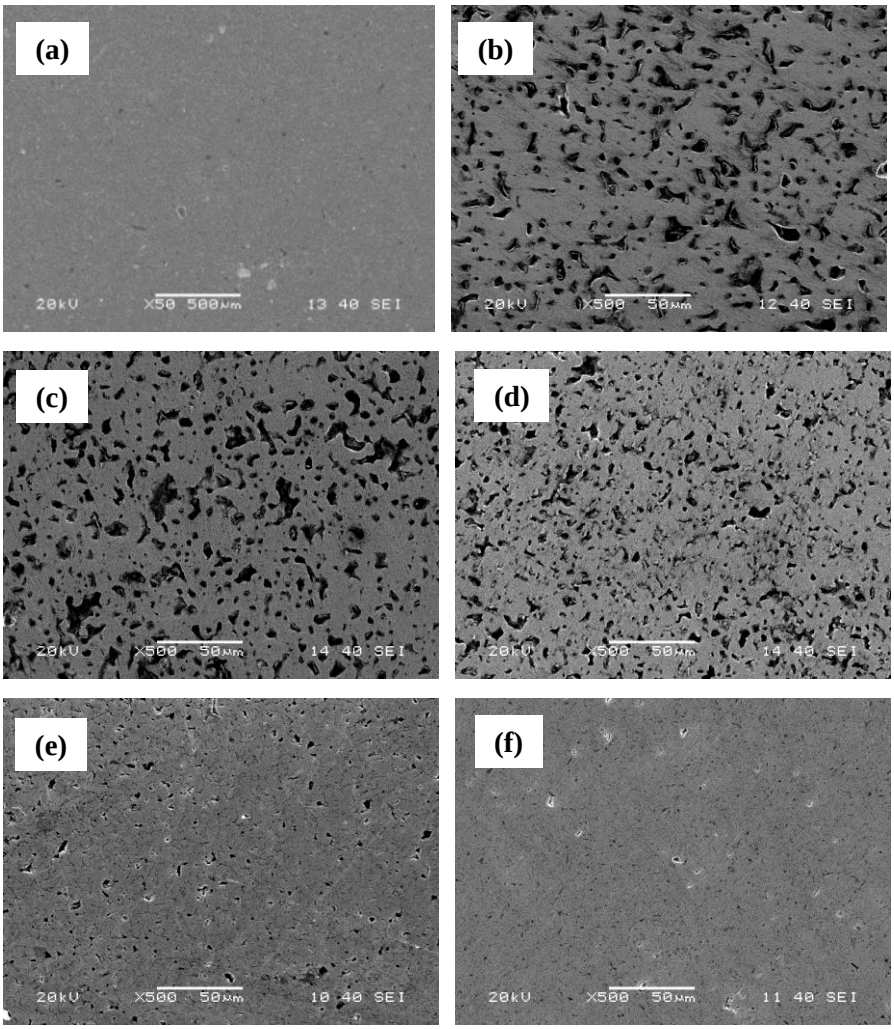


Figure 4.20 SEM image of Cu-graphite MMC's sintered at 900°C for 1h with different miiling time (a) 0h (b) 2h (c) 4h (d) 8h (e) 20h (f) 40h

4.2.3 Particle size measurements

Particle size of the copper and graphite powders were examined by Malvern laser particle size analyzer. Fig. 4.20 shows the variation in median size with milling time. It can be seen that median size increases at initial milling time (till 8h) due to flake formation and then decreases after 20h milling time due to strain hardening. SEM observation also supports the same finding (Fig. 4.19).

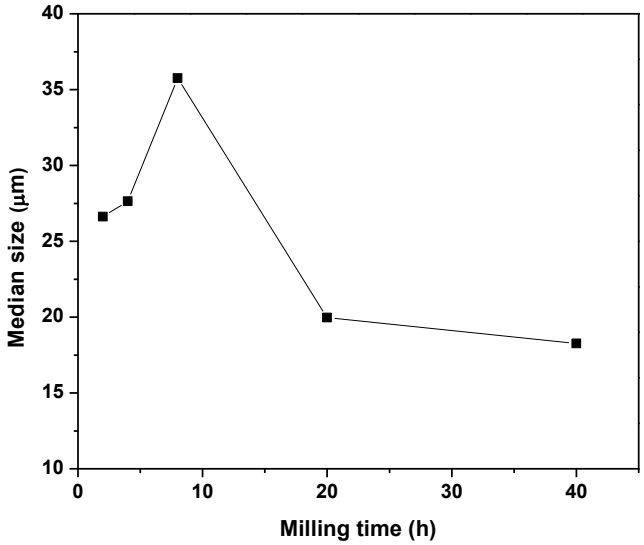


Figure 4.21 Variation of median size with vol. % of graphite

4.2.4 Density measurements

Fig. 4.22 shows the variation of density with milling time. It is found that density decreases up to 20 hours of milling and after that density increases. The presence of porosity leads to decrease in density up to 20 hours, after that porosity decreases and density goes up. SEM micrographs (Fig.4.20) also support the same observations as previously mentioned in section 4.2.2.

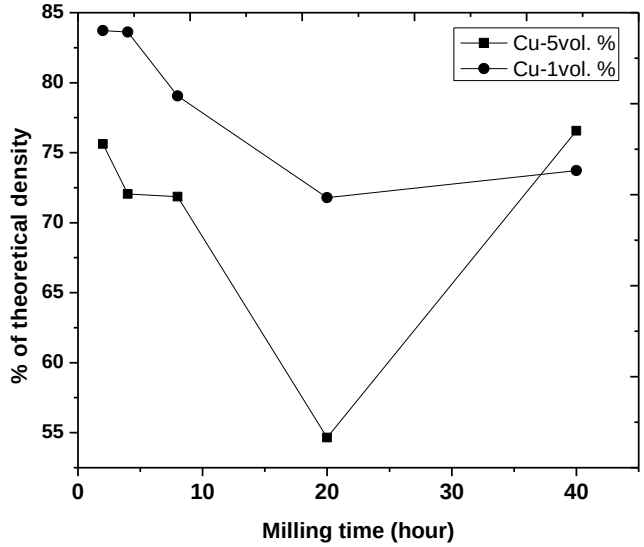


Figure 4.22 Variation of theoretical density with vol. % of graphite

4.2.5 Hardness study

Fig. 4.22 shows the variation in hardness with milling time. In the initial milling period (up to 10 h) hardness trend is decreasing but after that hardness value goes up. The reason is large porosity and less density in the initial milled samples and later porosity decreases and density increases. After 40 hours of milling, the particle becomes fine and more closely bound and particle-particle contact increases with increase in fineness due to increase in surface area of the milled powder particle. Another reason of increasing hardness is that Cu powder particles are coated with graphite powder to some extent and improves the bonding between Cu and graphite [21]. Guel et al. [23] studied the effect of metallic addition on mechanical properties of Al-graphite MMC by mechanical milling. Similar observations were made by Yusoff et al. [19].

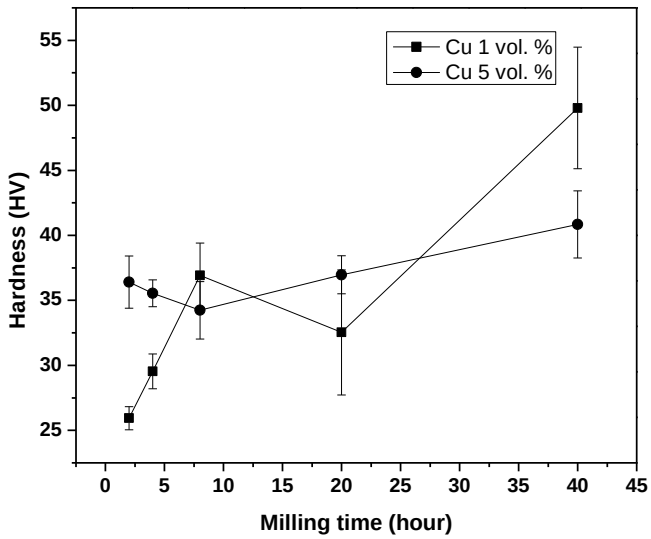


Figure 4.23 Variation of micro-hardness with vol. % of graphite

4.2.6 Wear study

Fig. 4.23 shows the variation of wear depth with milling time. Milling of initial Cu and graphite powder mixtures result in higher wear resistance than un-milled powder because with increase in milling time particle becomes fine, more closely bound, particle-particle contact increases and powder particles are activated. Sintering of such powder mixtures result in higher densification and hardening response than un-milled one.

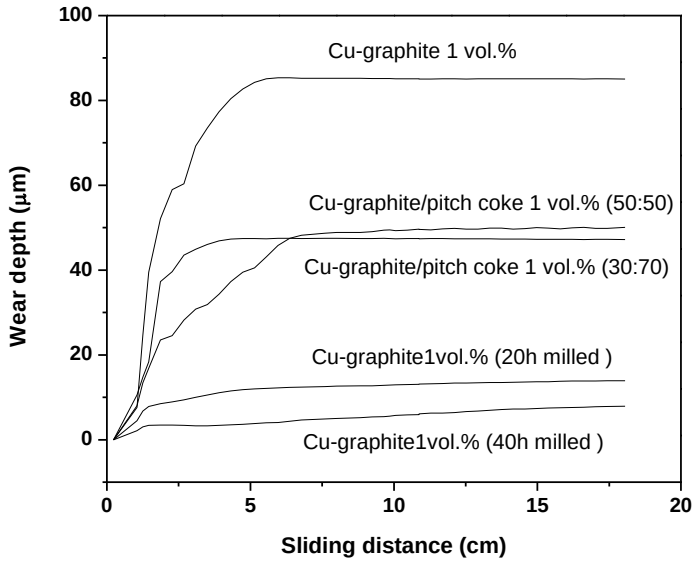


Figure 4.24 Variation of wear depth with sliding distance

4.2.7 Wear rate and wear volume

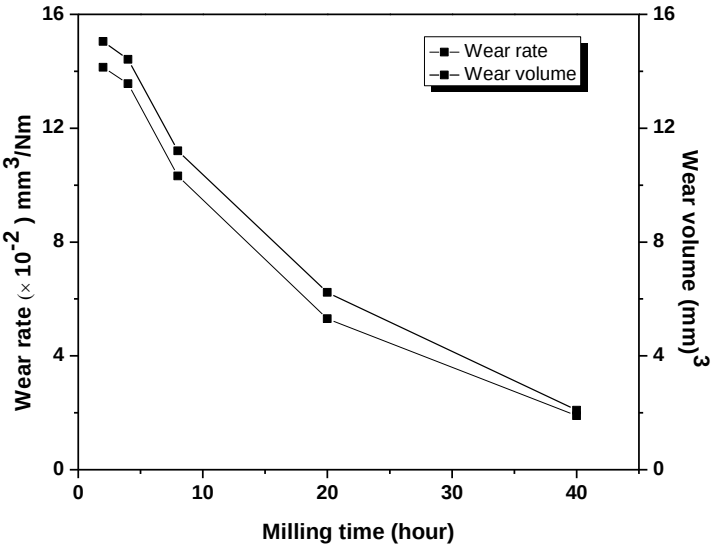


Figure 4.25 Variation of wear rate and wear volume with milling time

Wear volume and wear rate were calculated for Cu-graphite MMC's with variation in milling time. It is noticed that wear loss decreases with increase in milling time. Also wear volume and wear rate is lesser for Cu-graphite un-milled sample than milled.

4.2.8 Surface profile study

Fig. 4.25 shows surface profile of Cu-graphite MMC's milled for 40h which is measured from surface to wear track than to surface. It is also observed that surface roughness is less as compared to Cu-graphite/pitch coke MMC's because as the milling time approaches to 20h particle become fine and provide strong bonding and improved particle to particle. Higher hardness and wear resistance leads to low surface roughness.

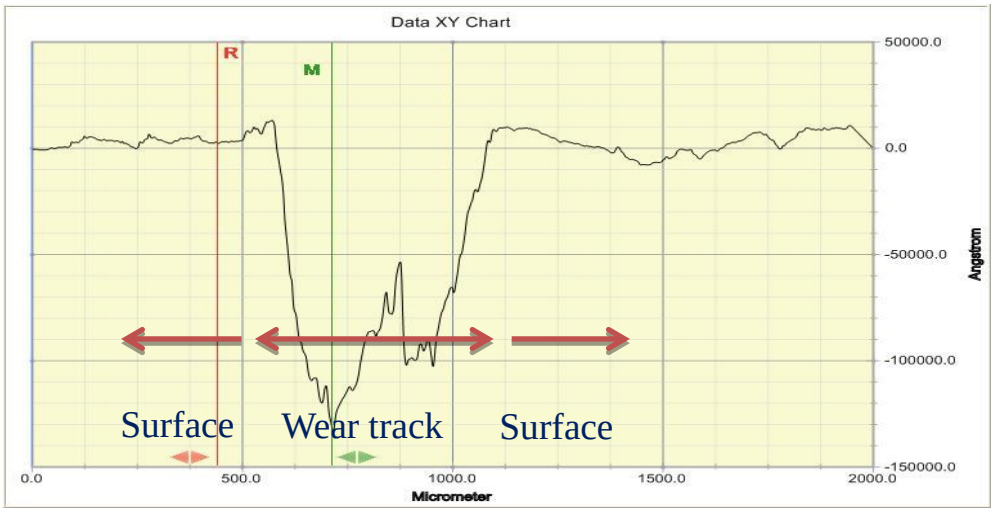


Figure 4.26 Surface profile of Cu-graphite MMC's (milled sample)

4.2.9 Electrical conductivity measurement

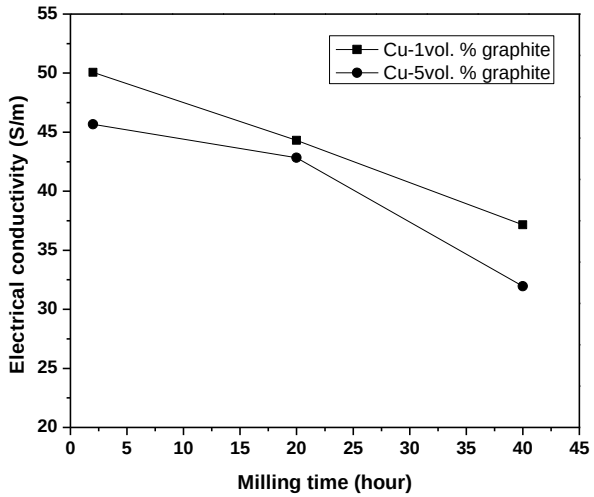


Figure 4.27 Variation of electrical conductivity as a function of vol. % of graphite

Fig. 4.27 shows the variation in electrical conductivity with milling time. The graph shows the decrease in electrical conductivity with milling time. As milling continues there is a decrease in grain size and as a results number of grain boundary increases. These grain

boundaries provide obstacles to electron movement and hence resistivity increases. Another reason is that during milling many defects and impurities are introduced into powder mixture and eventually resistivity increases. As the resistivity increase consequently conductivity decreases. It was also observed that for Cu-1 vol. % sample, electrical conductivity was less affected as compared to Cu-5 vol. % graphite because Cu-1 vol. % sample has very low amount of reinforcement (graphite) having lower electrical conductivity than Cu, leads to decrease in electrical conductivity.

Chapter 5

Conclusions

5 Conclusions

The following conclusions can be drawn from the present investigation.

1. The effect of pitch coke on wear and electrical conductivity of Cu-graphite composites has been studied systematically.
2. It has been found that graphite/pitch coke ratio of 30:70 provides higher hardness and wear resistance than the ratio of 50:50. It has also been observed that there is not much improvement on hardness due to soft nature of graphite.
3. The milling effect of Cu and graphite powder mixture on the fabrication of Cu-graphite MMC has been successfully studied. Initially, there is an increase in particle size due to flake formation as evident from SEM and particle size analysis. Further milling leads to strain hardening of powder and particle shape changes from flake to spherical.
4. Milling of initial Cu and graphite powder mixtures results in higher wear resistance than unmilled powder.
5. XRD study shows that no reaction takes place between copper and graphite. There is a presence of weak Cu_2O peak.
6. Wear resistance increases with increasing graphite content as it acts as lubricating film on the contact surfaces. Further addition of pitch coke into Cu-graphite composite powder mixture results in further reduction in wear depth.
7. Wear volume and wear depth also decreases with increasing graphite content and pitch coke.
8. Electrical conductivity decreases with increasing graphite, pitch coke content and milling time.

Chapter 7

Future work

6 Future work

1. The interface of the composites can be studied by using transmission electron microscopy (TEM).
2. Effect of co-efficient of friction on wear behavior.
3. Cu-graphite/pitch coke MMC can also be prepared by other advanced processing techniques such as spark plasma sintering, hot pressing, microwave sintering etc.

Chapter 6

References

7 References

1. William D. Callister “Material Science and Engineering” 7th edition ,John Wiley and Sons, University of Utah, (2007) pp. 577-578.
2. S. F. Moustafa, S.A. Elbadry, A. M. Sanad, B. Kieback “Friction and wear of copper–graphite composites made with Cu-coated and uncoated graphite powders” Wear (2002) pp. 699-710.
3. H. Yang, R. Luo, S. Han, M. Li “Effect of the ratio of graphite/pitch coke on the mechanical and tribological properties of copper–carbon composites” Wear 268 (2010) pp. 1337–1341.
4. F.C. Campbell “Introduction to composite materials” Structural Composite Materials, ASM international, Ohio 2010.
5. T. W. Chou, A. Kelly, A. Okura “fiber reinforced metal matrix composite” university of Delaware USA.
6. K.K. Chawla, N. Chawla “Composite Materials: Science & Engineering” 2nd edition, Springer-Verlag, New York, 1999.
7. H. Yang, R. Luo, S. Han, M. Li “Effect of the ratio of graphite/pitch coke on the mechanical and tribological properties of copper–carbon composites” Wear 268 (2010) pp. 1337–1341.
8. P. Queipo, M. Granda, R. Santamarıa, R. Menendez “Preparation of pitch based carbon copper composite for electrical applications” Fuel 83 (2004) pp. 1625-1634.
9. S.F. Moustafa, S.A. El-Badry, A.M. Sanad, B. Kieback “Friction and wear of copper–graphite composites made with Cu-coated and uncoated graphite powders" Wear 253 (2002) pp. 699-710.
10. J. Kovacik, S. Emmerb, J. Bielek , L. Kelesi “Effect of composition on friction coefficient of Cu-graphite” Wear 265 (2008) pp. 417–421.
11. F. Akhlaghi, A. Zare-Bidaki “Influence of graphite content on the dry sliding and oil impregnated sliding wear behavior of Al 2024–graphite composites produced by in situ powder metallurgy method” Wear 266 (2009) pp. 37-45.
12. X.C. Maa, G.Q. Hea, D.H. Hec, C.S. Chena, Z.F. Hua, “Sliding wear behavior of copper–graphite composite material for use in maglev transportation system” Wear 265 (2008) pp. 1087-1092.

13. B. Chen, Q. Bi, J. Yang, Y. Xia, J. Hao, "Tribological properties of solid lubricants (graphite, h-BN) for Cu-based P/M friction composites" Tribology International 41 (2008) pp.1145– 1152.
14. R.K. Gautam, S. Ray, Satish C. Sharma, S.C. Jain, R. Tyagi "Dry sliding wear behavior of hot forged and annealed Cu-Cr-graphite in situ composites" Wear 271 (2011) pp. 658-664.
15. H. Arami, A. Simchi "Reactive milling synthesis of Nano crystalline Al–Cu/Al_2O_3 Nano composite" material science and engineering 464 (2007) pp. 225-232.
16. A. Jain , B. Basu, B.V. Manoj Kumar, Harshavardhan , J. Sarkar "Grain size–wear rate relationship for titanium in liquid nitrogen environment" Acta Materials 58(2010) pp. 2313-2323.
17. Da Hai Hea, R. Manory, "A novel electrical contact material with improved self-lubrication for railway current collectors" Wear 249 (2001) pp.626–636.
18. O. Guler, E. Evin "The investigation of contact performance of oxide reinforced copper composite via mechanical alloying**"** Journal of Materials Processing Technology 209(2009) pp.1286-1290.
19. M. Yusoff, R. Othman, Z. Hussain, "Mechanical alloying and sintering of nanostructured tungsten carbide-reinforced copper composite and its characterization" Materials and Design 32 (2011) 3293–3298.
20. A. Fathy, F. Shehata, M. Abdelhameed, M. Elmahdy "Compressive and wear resistance of nanometric alumina reinforced copper matrix composites" Materials and Design 36 (2012) pp.100–107.
21. C.P. Samal, J.S. Parihar, D. Chaira "The Effect of Milling and Sintering Techniques on Mechanical Properties of Cu-Graphite Metal Matrix Composite Prepared by Powder Metallurgy Route" Journal of alloys and compounds 596(2013) pp. 95-101.
22. G. Parida, D. Chaira, M. Chopkar , A. Basu , "Synthesis and characterization of Ni-TiO_2 composite coatings by electro-co-deposition" Surface & Coatings Technology 205 (2011) pp. 4871–4879.
23. E. Guel, C. C. Gallardo, J.L.C. Cortés, E.R. Rangel, J.M.H. Ramírez, R.M. Sánchez, J. of Alloys Comp. 495 (2010) pp. 403–407.